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To: . Mr. J. E. Wickham

Date: January 26, 1990

From: . Jane Y. Lewis

Subject: . Accomplishments for the Analytical Chemistry Section - 1989

The accomplishments for the Analytical Chemistry Section are summarized below. Individual accomplishments for the professional staff are attached.

I. REPLACEMENT OF GAS CHROMATOGRAPHS IN THE NICOTINE AND WATER LABORATORY

A. Gas Chromatographs (J. Sampson, R. Forte)

Two Hewlett Packard 5890A capillary gas chromatographs for nicotine and water analysis were installed. These instruments are equipped with a 15-meter x 0.25 mm ID x 0.25 micron film thickness Stabilwax-DB capillary column for nicotine analysis and Poropak QS packed column for water analysis. An HP 5895A ChemStation provides the needed instrument and autosampler control as well as data collection and analysis. Investigation is currently underway with CAD to determine the best hardware and software configuration for the data acquisition system. (Memo from J. Sampson, "Proposed Systems for Nicotine and Water Laboratory," 4/10/89).

B. Sample Transfer Station (J. Sampson)

With the new Hewlett Packard gas chromatographs, there is a need to transfer TPM extraction solution from test tubes to autosampler vials for nicotine and water analysis. A Gilson Sample Changer was tested for this purpose and was proven to be very efficient in the transfer operation. The Gilson programs were modified to ensure no cross-contamination between samples.

This unit was also tested for the operation of filling test tubes with extraction solution. The operation is somewhat slower than the old Filamatic unit, but is more accurate in the volume dispensed and much more versatile in terms of dispensing different volumes. Having the ability to easily change volumes is advantageous when measuring ultra low delivery cigarettes or when nicotine/water analyses must be performed on a single port basis.

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II. SPECIAL SMOKING LABORATORY

A. Filtrona 20-port CO Smoking Machine (B. Joyner)

The Filtrona machine was installed to increase capacity for generating CO delivery in cigarettes. Data collected from CI brands as well as monitor cigarettes proved to be statistically not different from data generated on the Phipps & Bird machine. This work was summarized in a CTSD Project Review, 3/19/89 and in a memo entitled "Evaluation of Filtrona CO Smoking Machine," 6/2/89 by B. Joyner.

B. Air Handling System

A Liebert air handling system was installed for the purposes of obtaining ISO conditions (72°F, 60% RH) in this laboratory. These conditions are required for the testing of some international brands. The Liebert system also ensures consistent maintenance of FTC conditions (75°F, 60% RH). (Memo to J. E. Wickham from J. Sampson, "ISO Conditions - L-4217," 6/15/89.)

C. New 5-Port Smoking Machine (B. Joyner, J. Sampson)

The prototype system including new 5-port smoking machine and PC based data acquisition system were installed. Results from monitor cigarettes for TPM, puff count, CO and NO delivery are within specifications. The TPM values from each port were plotted on control charts to show that there was no bias from port-to-port. This machine has been put into routine operation.

D. Cyanide Delivery (J. Sampson)

The level of total cyanides in Monitor #25 cigarettes were below specifications. Attempts to bring delivery within specification included investigation of air flows, smoking machine operation, quality of house air and water, efficiency of transfer from gas phase to aqueous phase and operation and performance of the autoanalyzer used for detection. Bill Harvey, Cindi Bright, and Becky Kanipe were consulted for their expertise in this area. Efforts thus far have failed to correct the situation. The installation of the new air handling system which provides a very consistent supply of conditioned air may effect a difference. (Memo from J. Sampson, "Gas Phase Laboratory," 6/9/89. Memo from B. Kanipe, "Troubleshooting Summary of Hydrogen Cyanide in Gas Phase," 11/16/89.)

E. Carbon Filtered Cigarettes (J. Sampson)

Smoke Index data were generated for carbon filtered cigarettes with and without the ventilation holes taped to see if small differences in delivery could be magnified. This work was proposed in a memo to J. E. Wickham from J. Y. Lewis entitled "Gas Phase Methodology for Comparing Plasticizers in Carbon Filters," 6/1/89. The data showed that trends between taped and untaped cigarettes were consistent and that the taped experiment offered

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no additional information. These results were summarized in a CTSD Project Review by J. Sampson, 12/7/89.

III. ALKALOID REDUCED TOBACCO CIGARETTES

A. U.S. Testing Certification (R. Forte)

It was requested of U.S. Testing Company, Inc., to certify the capillary gas chromatographic method for quantifying low levels of nicotine in smoke for ART cigarettes. Cigarettes at the level of 0.04 and 0.07 mg nicotine were analyzed as is, and with small known quantities of nicotine added. Recoveries averaged 96-107% for the spiked additions. An extraction study proved an 1-hour extraction time to be sufficient. This method was certified in a report by Boyd Fagan of U.S. Testing entitled "An Improved Technique for Accurate Measurement of Low-Level Nicotine Delivery in Cigarette Smoke," 9/13/89.

B. Wide Bore vs. Capillary Chromatography (J. Sampson)

Objective: To evaluate capillary versus wide-bore columns for nicotine analysis on ART smoke samples.

Status: A study was completed which showed excellent agreement between a 15-meter x 0.25 mm ID x 0.25 micron film thickness Stabilwax-DB capillary column and a 15-meter x 0.53 mm ID x 1.00 micron film thickness Stabilwax-DB wide-bore column. The wide-bore column was installed in an HP5880A packed column GC using HP injector port adapters and glass liners. A 5-meter fused silica guard column was installed before the analytical column which allowed the analysis time to be reduced from 40 to 20 minutes per sample. Although all ART smoke samples are currently being analyzed on a capillary GC, if a capillary GC is not available, nicotine can be analyzed on a packed column GC equipped with wide-bore columns. This work was discussed in a CTSD Project Meeting by J. Sampson, 3/28/89 and in a memo from J. Sampson entitled "Nicotine Analysis," 3/13/89. The wide bore methodology was documented in a completion report entitled "Determination of Nicotine in Smoke for Alkaloid Reduced Tobacco," by J. Y. Lewis, R. A. Forte and J. A. Sampson, Accession No. 89-036, 7/21/89.

C. Quality Assurance (J. Sampson)

A Hewlett Packard 5880 gas chromatograph was set-up in QA Product Audit for the analysis of nicotine in ART products. The column used was a 15-meter x 0.25 mm ID Stabilwax-DB capillary column. (Memo from J. Sampson, "Capillary Gas Chromatograph for QA," 3/9/89.)

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IV. ROBOTIC DISPENSING SYSTEM (R. Forte)

The robotic system was designed and built by Hudson Robotics, Inc. to meet our detailed criteria for operating and safety features. The system dispenses ethanolic extraction solvent onto samples for the analyses of menthol, humectants and plasticizers. The system can accommodate both the test tubes and Erlenmeyer-type disposable flasks that are used in this laboratory. It can be programmed to process a large number of samples with several different volumes of extraction solution. An extensive set of safety features were incorporated in order to obtain Factory Mutual's approval (PM's fire insurance carrier). This work was documented in a completion report entitled "Laboratory Robotic Dispensing System," by R. A. Forte, R. W. Kanipe and J. Y. Lewis, Accession No. 89-042, 6/6/89. It was also presented at TCRC by R. A. Forte, Richmond, Virginia, 10/4/89.

V. PEAK COAL TEMPERATURE (B. Joyner)

A thermographic image analysis system was purchased from Agema Infrared to measure the peak coal temperature of a burning cigarette. This was replacement of a 20-year old AGA system that had become inoperative. Customized software was provided by Agema so that data could be captured automatically from ten cigarettes being smoked on a rotary-type smoking machine. The peak coal temperature data are reported on a per puff and per cigarette basis. The system is fully automated and designed for high volume usage.

Data from monitor cigarettes are comparable to that produced on the AGA system. The Agema system has many features and advantages that were not available in the past. An overview of the system was presented by B. Joyner and D. Lisbon at the CTSD Project Review, 12/7/89.

VI. MENTHOL/HUMECTANTS (J. Sampson)

The HP 5880 gas chromatograph used for menthol in filler, and humectants in smoke and filler was modified to improve efficiency. The analysis is performed on a 30-meter x 0.53 mm ID DB-WAX wide-bore column with an analysis time of 45 minutes per sample. The installation of a five-meter x 0.53 mm ID fused silica guard column before the analytical column resulted in a decrease in analysis time to 25 minutes per sample. (Memo from J. Sampson, "Humectants Analysis," 4/18/89.)

VII. ANETHOLE ANALYSIS IN FILLER AND AFTERCUT (R. Forte)

A dual distillation apparatus was assembled and put into operation for extracting anethole. The required capillary gas chromatograph is being installed. In the meantime, sample preparation is being done in CTSD with chromatographic analysis in Flavor Development. Following collaborative efforts between the two laboratories, CTSD will accept full responsibility for quantifying trace level (1-5 ppm) anethole in filler and aftercut was transferred from Flavor Development to CTSD.

VIII. CITRATES IN PAPER BY ION CHROMATOGRAPHY (B. Joyner)

An ion chromatographic procedure as replacement for gas chromatography was adapted for CTSD's use by Garland Carter. The ion chromatographic procedure eliminates the use of chloroform as an extraction solvent. The method was then transferred to the Analytical Chemistry Section. One remaining chemical interference needs to be investigated before the method can be put into routine use.

IX. DIN METHODOLOGY (R. Forte)

A procedure was developed for quantifying nicotine and water in TPM of samples smoked according to the DIN procedure. TPM was received in an electrostatic precipitation tube from the Borgwaldt smoking machine. Several samples were analyzed to establish a correlation of tar and nicotine between the DIN and FTC methodologies.

X. REPORTS

- A. "Humectants and Plasticizer Levels of Commercial Cigarettes - 1988," memo to J. E. Wickham from R. A. Forte, 3/31/89.
- B. Annual Freon® Survey, reported by B. Joyner in CTSD August, 1989 monthly report, 8/89.

XI. TRAINING

B. Joyner and J. Sampson spent time in September, 1989 training Mr. Yap Swee Chye, chemist for the Department of Scientific Services in Singapore. His training within the Analytical Chemistry Section included operation and maintenance of the Filtrona 20-port smoking machine and Hewlett Packard 5890 gas chromatograph. He was also trained in the preparation of nicotine and water standards and in data evaluation.

XII. MEETINGS

- A. IRI Management Study Group, Baltimore, MD, 1/9-11/89.
- B. Pittsburgh Conference, Atlanta, GA, 3/6-10/89.
- C. International Conferences on Fourier Transform Spectroscopy, Fairfax, VA, 6/19-21/89.
- D. TCRC, Richmond, VA, 10/3-4/89.
- E. Priority Management, in-house, 11/16/89.

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